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Uranium stripping from tri-*n*-butyl phosphate by hydrogen peroxide solutions

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ABSTRACT

The processes of uranium stripping from 30% tri-*n*-butyl phosphate (TBP) in "odorless" kerosene by H_2O_2 solutions both with and without NH_4OH added were investigated in the temperature range of 20–50 °C and with a volumetric ratio of 1 between the organic and aqueous phases. The uranium was selectively precipitated in the form of uranium peroxide during stripping from the organic phase by hydrogen peroxide. The stripping of uranium increased with increasing H_2O_2 content, increasing temperature and increasing concentration of NH_3 in the range of 0-15 g/L. The use of a heated solution (40 °C) that contained 4 mol H_2O_2/mol U and NH_3 12 g/L resulted in 99.7% of the uranium being stripped from TBP in the form of uranium peroxide. The uranium peroxide obtained by stripping is a highly pure product that exists in two different hydrated forms: $UO_4{\cdot}4H_2O$ (92 mass %) and $UO_4{\cdot}2H_2O$. The mean particle diameter was 20.75 μm. The effect of hydrogen peroxide on the organic phase was studied by IR spectroscopy. No structural changes in TBP were observed after 30 cycles of extraction/stripping; thus, the use of hydrogen peroxide in this application is unrestricted.

1. Introduction

Uranium concentrate obtained at a manufacturing plant in the form "yellow cake" or calcined yellow cake represents only the first step in the processing of uranium ore toward the production of enriched uranium (Hausen, 1998; Morss et al., 2010). Although the uranium has been separated from the majority of impurities present in the ore during the mining and milling operations, yellow cake is not a high-purity product and is normally further refined to produce nuclear-grade material (Morss et al., 2010; Wilson, 1996). Specifically, the uranium must be separated from elements with high thermal-neutron capture cross-sections and from other impurities that form volatile fluorides (Wilson, 1996). The uranium purification is performed either by solvent extraction from nitric acid solutions or by distillation of uranium hexafluoride (Edwards and Oliver, 2000; Morss et al., 2010). Solvent extraction is currently the most common method for uranium refining circuits. This process has been well described in the literature (Edwards and Oliver, 2000; Morss et al., 2010; Sato, 1958; Wilson, 1996). It consists of the dissolution of yellow cake in nitric acid and the selective extraction of uranium using tri-*n*-butyl phosphate (TBP). The purified uranium is usually stripped from the organic phase into the aqueous phase by water or diluted nitric acid. The product of uranium stripping (i.e., uranyl solution) is subsequently evaporated to a molten salt of uranyl nitrate. The molten uranyl nitrate is converted to UO_3, reduced with hydrogen to UO_2, and converted to UF_4 with hydrogen fluoride at elevated temperatures (Morss et al., 2010).

This work describes the application of hydrogen peroxide as an alternative agent for the stripping of uranium from loaded TBP. This method differs from traditional methods that employ water (or diluted nitric acid) for stripping the metal from the organic phase. In fact, uranium is stripped from the loaded organic phase through direct precipitation in the form of uranium peroxide. The use of H_2O_2 for the precipitation of uranium as uranium peroxide has been mentioned in a number of publications. However, all of them describe a process of uranium precipitation from acidic solutions (e.g., mill solutions, in situ leach liquors, eluted solutions, and pregnant strip solutions) (Cahill and Burkhart, 1990; Caropreso and Kreuz, 1977; Gupta et al., 2004; Hardwick, 1984; Morais et al., 2005; Shabbir and Tame, 1974; Singh et al., 2002). This paper describes our research on the stripping of uranium from a loaded organic phase using hydrogen peroxide solutions. This process has several advantages (e.g., a product with greater purity, reduced volume of waste solutions, easy handling, etc.) and can lead to the elimination of some processing steps of uranium refining, such as the expensive operation of evaporation and unprofitable denitration stage.

2. Experimental

The loaded organic phase containing 78 g/L U(VI) and 15.34 g/L HNO_3 was prepared through the selective extraction of uranium from uranyl nitrate solution (120 g/L U(VI), 100 g/L HNO_3) using 30%

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(by volume) TBP in "odorless" kerosene. The extraction was conducted in a separatory funnel at room temperature (20–25 °C). The volumetric ratio between the organic and aqueous phases (O/A) was equal to 1. The uranyl nitrate solution was prepared by the dissolution of ammonium polyuranate in nitric acid solution. Ammonium polyuranate was obtained from the ore uranium concentrate. The content of impurity elements in the uranyl nitrate solution was equal to (mg/L): 228 Fe, 204 S, 7.6 K, 11.2 Na, 3 Mo, 0.46 V, 0.07 W, 192 Si, 72 P, 5.9 Cr, 0.192 B, 19.2 Cl, 40.8 Ti, 94.8 Al, 0.36 Cu, 1.2 Pb, 3.36 Ni, 6.24 Mn, 0.216 Li, 4.8 Mg, and 5.8 Ca. All other chemicals used were reagents of chemically pure grade.

The stripping procedures were performed in a separatory funnel (100 mL) at 20–50 °C. In all the experiments, the volumetric ratio between the organic and aqueous phases (O/A) was equal to 1. The uranium was stripped from the loaded organic phase by the addition of hydrogen peroxide, and the pH of the reaction was controlled through the addition of 5% ammonium hydroxide solution. The hydrogen peroxide solutions containing 1–30 mol H_2O_2 for each mol of uranium in the organic phase were prepared by the dilution of 30% hydrogen peroxide shortly prior to experiments. Distilled water was used in the dilutions.

The stripping experiments were performed using the following procedure. Equal volumes (20 mL) of loaded organic phase and hydrogen peroxide solution with or without NH_4OH added were placed in a separatory funnel and were rapidly stirred. The phases were stirred 2 min in all of the experiments. The phases were allowed to stand alone prior to the precipitation of uranium peroxide (0.15–24 h). All three phases were subsequently separated.

The uranium concentration in the aqueous phase was determined using ICP-AES (Optima 2100 DV Perkin Elmer). The nitric acid content in the aqueous phase was determined by acid–base titration with methyl orange as an indicator. The analysis of uranium peroxide obtained by stripping was performed using ICP-AES. The uranium concentration and the HNO_3 concentration in the organic phases were calculated on the basis of the mass balance.

X-ray phase analysis of uranium peroxide powders was performed at STOE STADI-P. All infrared spectra of the organic phases and uranium compounds were measured in the range of 400–4000 cm^{-1} using a Bruker VERTEX 70 spectrometer. The measurements of IR spectra of uranium peroxide were performed on solid-phase samples using the KBr pellet technique. The mixtures of KBr and the samples were pressed into transparent pellets (d = 13 mm) at 600 MPa. IR spectra of the organic phases were obtained by placing one drop of a pure sample between polyethylene plates. The particle size distribution of uranium peroxide was determined using an Analysette 22 laser particle sizer. The thermogravimetric analysis (TGA) and differential thermal analysis (DTA) were performed on a Mettler Toledo TGA/SDTA 851e using a heating rate of 10 °C/min.

All the experiments were performed at least in duplicate, and the average error was less than 1.0%.

3. Results and discussion

The use of a hydrogen peroxide solution for stripping from loaded organic phases results in the precipitation of uranium in the form of uranium peroxide, as shown in Eq. (1):

$$UO_2^{2+} + H_2O_2 + nH_2O = UO_4 \cdot nH_2O + 2H^+ \quad (1)$$

It is known that the use of excess of hydrogen peroxide, the pH, the precipitation time and the temperature are importance known to be important in the production of uranium peroxide (Gupta et al., 2004; Kim et al., 2011). Therefore, in order to find optimal conditions for uranium stripping, the effects of the temperature, the concentrations of content of H_2O_2 and NH_3 in the stripping solution, and the holding time of the phases were studied. The effect of the hydrogen peroxide on the organic phase was studied by IR spectroscopy.

3.1. Effect of H_2O_2 content on the uranium stripping

According to Eq. (1), one mol of H_2O_2 is required for each mol of uranium. However, the excess of hydrogen peroxide is known to be required for the precipitation of uranium from solutions (Cahill and Burkhart, 1990; Gupta et al., 2004; Morais et al., 2005). This necessary excess may vary according to the uranium solution composition (Morais et al., 2005).

The effect of the H_2O_2 content on the uranium stripping was studied at room temperature (20–25 °C). The uranium was stripped from the loaded organic phase using hydrogen peroxide solutions (1–30 mol H_2O_2/mol U). All other parameters were kept constant in these tests. The results of these experiments are presented in Fig. 1. The obtained results indicated that the uranium stripping increases as the H_2O_2 content in the solution increases. A large excess of hydrogen peroxide is required for almost complete uranium stripping; therefore, the use of pure H_2O_2 solution for stripping is economically unviable.

The separation time of phases was also observed to decrease as the amount of excess H_2O_2 was increased due to an increase in the particle size of uranium peroxide.

3.2. Effect of NH_3 concentration on the uranium stripping

The final pH of the aqueous phase was 0.16–0.28 after stripping of uranium from TBP by using H_2O_2. Uranium peroxide is known to precipitate from solutions in a pH range of 2.5–4.0 (Cahill and Burkhart, 1990; Gupta et al., 2004; Morais et al., 2005; Morss et al., 2010; Watt et al., 1950). Therefore, diluted ammonia (5%) was added to the stripping solution to maintain the desired pH for uranium peroxide precipitation. The experiments were performed using a stripping solution that contained 1–4 mol H_2O_2/mol U and 0–15 g/L NH_3. The phase contact time was 0.15 h after stirring was stopped. The final pH of the aqueous phase was recorded after the phases separated. The results are shown in Fig. 2. The extent of uranium stripping was found to increase as the final pH of aqueous phase increased. The ammonia concentration in the stripping solution should be 12–15 g/L (i.e., the final pH of the aqueous phase should be in the range 2.7–3.3) to achieve efficient uranium peroxide precipitation (98%). Some part of the uranium was observed to precipitate as ammonium uranate at NH_3 concentrations greater than 15 g/L (i.e., when the final pH of the aqueous phase was greater than 3.3).

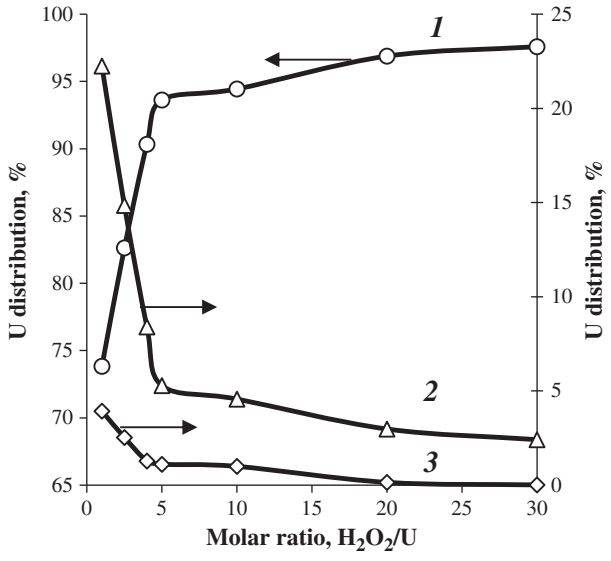

Fig. 1. The effect of H_2O_2 content in solution on uranium distribution during stripping from loaded organic phase: 1 – uranium peroxide; 2 – organic phase; 3 – aqueous phase.

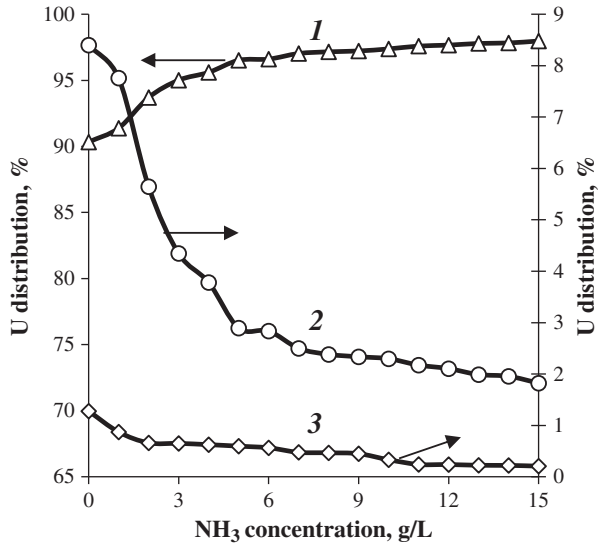

Fig. 2. The effect of NH_3 concentration on uranium distribution during stripping from loaded organic phase by solution containing 4 mol H_2O_2/mol U: 1 – uranium peroxide; 2 – organic phase; 3 – aqueous phase.

3.3. Effect of temperature on the uranium stripping

Uranium was stripped from the loaded organic phase at different temperatures (20–50 °C) with the other parameters held constant. Tests were performed using a solution that contained 4 mol H_2O_2/mol U and 12 g/L NH_3. The phase contact time was 0.15 h after stirring was stopped. The results are shown in Fig. 3. The uranium stripping was found to increase as the temperature was increased in the range of 20 to 40 °C and to decrease at temperatures greater than 40 °C. The decrease at temperatures greater than 40 °C occurred because the hydrogen peroxide decomposed more rapidly at these temperatures. Thus, when the temperature is increased, additional H_2O_2 is required for uranium stripping. The amount of uranium stripped from the loaded organic phase was maximal at a temperature of 40 °C. In this case, 98.8% of uranium was stripped in the form of uranium peroxide precipitate.

3.4. Effect of holding time on uranium stripping

The effect of the phase contact time after stirring (i.e., the holding time) on uranium stripping was studied using 0.2 L of the loaded organic phase. The stripping studies were performed in a separatory funnel (500 mL) at temperatures from 20 to 40 °C using H_2O_2 solutions (1–4 mol H_2O_2/mol U) both with and without NH_4OH added (12 g/L NH_3). Samples of the organic and aqueous phases were drawn hourly, and their uranium content was analyzed. The results indicate that the amount of uranium stripped from the loaded organic phase increases as the holding time is increased. The uranium stripping was almost complete within 10–24 h (Fig. 4). However, 99.7% of the uranium was precipitated from the organic phase after a holding time of 3 h at a temperature of 40 °C when the stripping solution contained 4 mol H_2O_2/mol U and 12 g/L NH_3. The final pH of the aqueous phase was 3.3. For all subsequent studies, these parameters were accepted as the optimal conditions for uranium stripping.

3.5. Effect of hydrogen peroxide on the organic phase

TBP is known to be sufficiently chemically and radiation stable (Barelko et al., 1966). TBP is well able to withstand physical destruction and chemical oxidation. However, under certain conditions, TBP is decomposed to dibutyl phosphate (DBP) and monobutyl phosphate (MBP), which form stronger complexes with certain impurity metal ions and promote their extraction from an aqueous solution into an organic phase (Barelko et al., 1966; Pai et al., 1982). Hydrogen peroxide is a strong oxidant. Therefore, the effect of H_2O_2 on TBP was studied by IR spectroscopy. Fig. 5 shows the spectrum of 30% TBP in odorless kerosene before extraction (curve 1) and after uranium was stripped under the optimal conditions previously described (curve 2). The IR spectrum shows the following major regions: a weak broad band in the 400–550 cm^{-1} region due to the deformation of P = O; an asymmetric and a symmetric bending of the ONO group at 669 and 722 cm^{-1}, respectively; weak bands at 735 and 771 cm^{-1}, which are assigned to CH_2 groups; small bands at 867 and 959 cm^{-1}, which are assigned to the symmetric and asymmetric stretching vibrations of the uranyl group, respectively; a C–C stretching vibration band at 911 cm^{-1}; stretching vibrations of the P–O–(C) group at 992, 1029 and 1061 cm^{-1}; bands at 1120 and 1151 cm^{-1}, which are assigned to the stretching vibrations of the C–O–(P) group; stretching

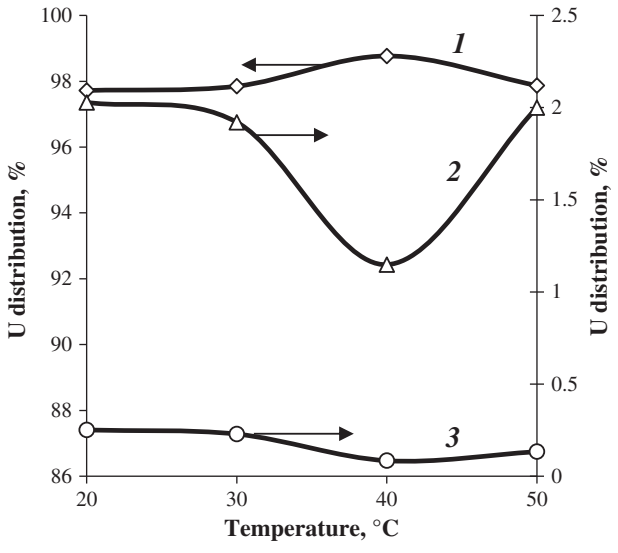

Fig. 3. The effect of temperature on uranium distribution during stripping from loaded organic phase by solution containing 4 mol H_2O_2/mol U and 12 g/L NH_3: 1 – uranium peroxide; 2 – organic phase; 3 – aqueous phase.

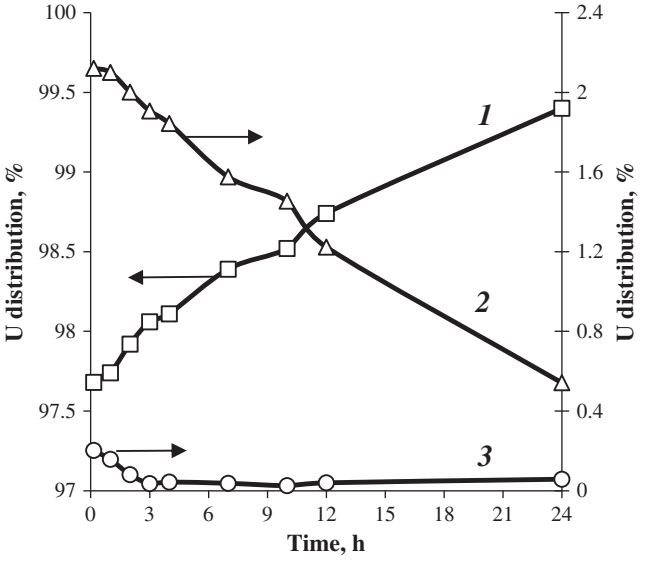

Fig. 4. The effect of holding time on uranium distribution during stripping from loaded organic phase by solution containing 4 mol H_2O_2/mol U and 12 g/L NH_3: 1 – uranium peroxide; 2 – organic phase; 3 – aqueous phase.

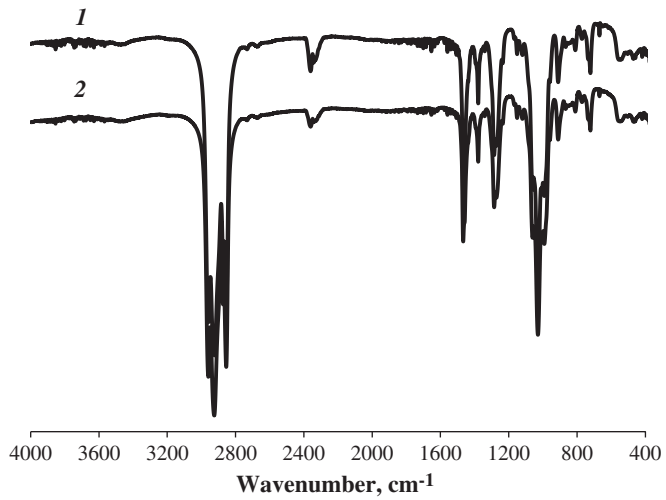

Fig. 5. Infrared spectra for organic phase: 1 – before uranium extraction; 2 – after uranium stripping by hydrogen peroxide solution.

vibrations of the P = O group at 1236, 1272 and 1285 cm^{-1}; bands in 1350–1500 cm^{-1} region, which are characterized by the bending vibrations of CH_3 and CH_2 groups; and bands in the 2800–3000 cm^{-1} region due to the symmetric and asymmetric stretching vibrations of CH_3, CH_2 and CH groups. The IR bands assignments are consistent with those in the literature (Bagnall and Wakerley, 1975; Borkowski et al., 2002; Ohwada, 1968; Piras, 2002; Sato, 1965). The peak in 2250–2550 cm^{-1} region is assigned to uncompensated interference due to carbon dioxide in the atmosphere of the sample compartment Fig. 5 and Fig. 9). According to the results of the investigations, structural changes in the organic phase were not observed after 30 cycles of extraction/stripping. The bands of MBP, DBP and other third phases were not observed in the spectra. Thus, hydrogen peroxide can be used for uranium stripping without restriction.

3.6. Properties of uranium peroxide

In all the experiments, the uranium peroxide precipitates were filtered, washed and dried in air at room temperature (20–25 °C). The precipitated uranium peroxide was studied using various methods. Figs. 6–9 and Table 1 show the results of the characterization of the

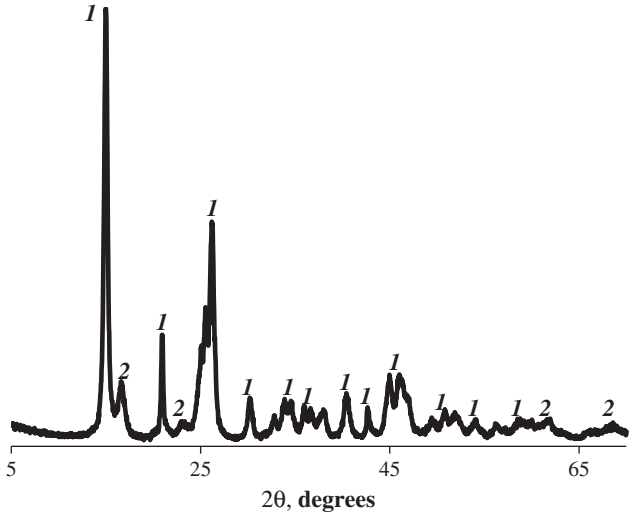

Fig. 6. X-ray powder diffraction pattern of uranium peroxide: 1 – $UO_4{\cdot}4H_2O$; 2 – $UO_4{\cdot}2H_2O$.

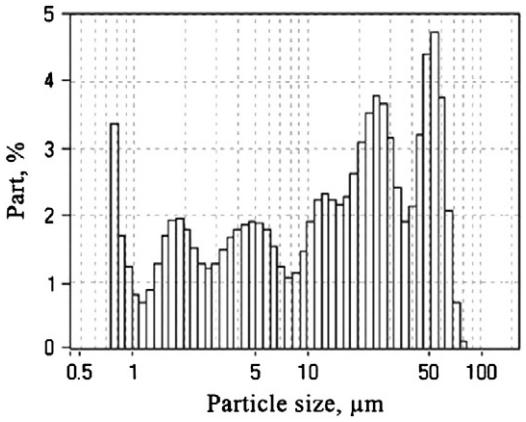

Fig. 7. Particle size distribution of uranium peroxide.

uranium peroxide that was precipitated from the loaded organic phase under the previously described optimized conditions.

As a result, uranium peroxide exists in two different hydrated forms: $UO_4{\cdot}4H_2O$ and $UO_4{\cdot}2H_2O$ (Fig. 6). According to the results of the analyses, the basic phase of uranium peroxide hydrate is the tetrahydrate phase (approximately 92 mass%). This result is in agreement with the results of previous studies of uranium peroxide precipitation from aqueous solutions of uranyl nitrate with hydrogen peroxide (Sato, 1961; Sato, 1963). The particle size distribution of the product is shown in Fig. 7. The particle mean diameter is 20.75 μm. The results of the chemical analyses of the uranium peroxide are given in Table 1. Analysis of the data leads to the conclusion that the uranium peroxide obtained by stripping is a high-purity product. The U_3O_8 content in the product is 90.14%.

The thermal decomposition of the uranium peroxide precipitate was investigated by TGA, DTA and infrared spectroscopy. The TGA and DTA curves are shown in Fig. 8. The DTA curve shows three endothermic peaks: the decomposition of $UO_4{\cdot}4H_2O$ to $UO_4{\cdot}2H_2O$ at approximately 130 °C, the decomposition of $UO_4{\cdot}2H_2O$ to hydrated UO_3 at approximately 245 °C and the conversion of UO_3 to U_3O_8 at approximately 628 °C. These reactions occur at points near the change in shape of the TGA curve. In addition, the TGA curve shows a small loss in weight at approximately 383 °C. This weight loss is due to the dehydration of hydrated UO_3. The TGA/DTA data is in good agreement with the infrared spectroscopy results. Infrared spectra of the uranium peroxide and the thermal decomposition products are shown in Fig. 9. The infrared spectrum of uranium peroxide powder, which was recorded at room temperature (22 °C), is shown in Fig. 9 (1). This spectrum contains several absorption bands with maxima at 3500, 3152, 1624, 1384, 907, 728 and 469 cm^{-1}. The two broad bands with maxima at 3500 and 3152 cm^{-1} are due to the

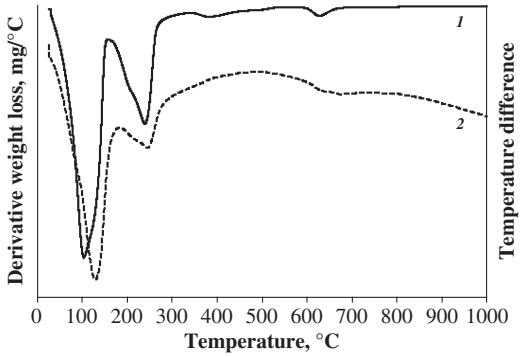

Fig. 8. TGA (1) and DTA (2) curves for uranium peroxide.

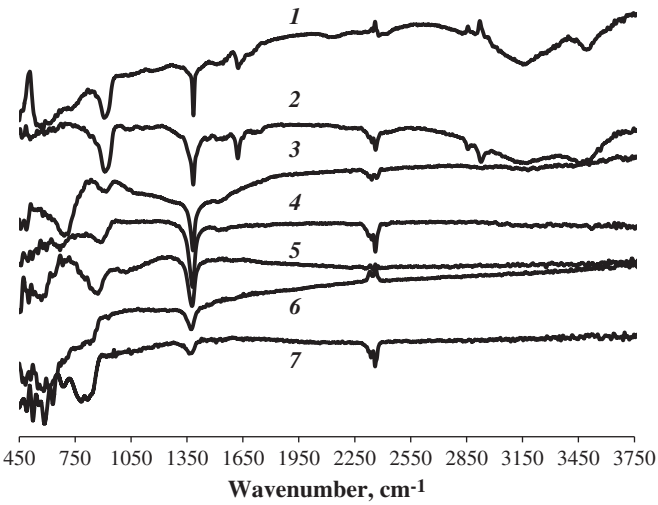

Fig. 9. Infrared spectra for uranium peroxide at different temperatures: 1 – 22 °C; 2 – 100 °C; 3 – 200 °C; 4 – 300 °C; 5 – 400 °C; 6 – 500 °C; 7 – 600 °C.

O–H stretching vibrations. The sharp peak at 1624 cm^{-1} is attributed to the vibrational bending mode of H_2O. The absorption band at 907 cm^{-1} is due to the asymmetric vibrations of the uranyl group. The band at 728 cm^{-1} is attributed to the vibration of H_2O groups. The highly intense band at 1384 cm^{-1} is due to the symmetric deformation of NH_3; this band is probably due to the presence of a NH_4^+ impurity. The band at approximately 469 cm^{-1} is assigned to U — O stretching. IR spectra of uranium peroxide heated at increasing temperatures in the range of 100 to 600 °C are shown in Fig. 9 (2–7). In the 22–200 °C range, new bands at 400–800 cm^{-1} appeared in the spectrum due to the decomposition of $UO_4{\cdot}4H_2O$ to $UO_4{\cdot}2H_2O$. At 100 °C, the OH-stretching band at 3152 cm^{-1} split into two bands at 2927 and 3158 cm^{-1}. Between 200 and 300 °C, the OH-group bands disappeared completely, whereas new bands appeared at 681 and 892 cm^{-1} due to the U–O stretching vibration of UO_3. The intensity and position of these bands changed at 300–500 °C. In the 500–600 °C range, the intensity of the band at 1384 cm^{-1} decreased. At 600 °C, a strong band appeared at 800 cm^{-1} due to the asymmetric stretching frequency of the uranyl group of U_3O_8. These TGA/DTA and IR spectroscopy results are consistent with the results of previous research reported in the literature (Cordfunke and Aling, 1963; Deane, 1961; Hoekstra and Siegel, 1961; Sato, 1976; Singh et al., 2004; Varga et al., 2011).

All of the results of the studies of the other precipitates obtained under different conditions were similar to those previously described, although they are not presented in this paper.

4. Conclusions

The results of this work clearly indicate that uranium can be effectively stripped from a loaded organic phase using hydrogen peroxide. The use of a hydrogen peroxide solution for stripping results in the precipitation of uranium in the form of uranium peroxide, and 99.7% of the uranium was precipitated as uranium peroxide from a loaded organic phase that contained 78 g/L U(VI) and 15.34 g/L HNO_3 under the following conditions: O/A = 1; stripping solution – H_2O_2 content 4 mol/mol U, NH_3 content 12 g/L; holding time 3 h; final pH of aqueous phase 3.3; stripping temperature 40 °C. The uranium peroxide obtained under these conditions is a highly pure product with a mean particle diameter of 20.75 μm. The U_3O_8 content in the product is 90.14%. We found that the basic phase of uranium peroxide is $UO_4{\cdot}4H_2O$. The hydrogen peroxide was found to not affect the organic phase; thus H_2O_2 can be used unrestricted.

The uranium peroxide obtained in this work was converted to UF_4. According to previous studies, this UF_4 can be used to produce UF_6 that corresponds to ASTM C787-11.

Table 1
Impurity elements in uranium peroxide ($\times 10^{-3}$ %), nf: not found.

Fe	4.1	Mo	0.09	Al	nf
K	0.9	V	nf	Cu	nf
Na	4.0	Si	nf	Pb	nf
S	15.8	P	3.7	Ni	nf
Ca	nf	B	0.02	Mn	0.6
Cr	nf	Cl	nf	Li	nf
W	0.01	Ti	nf	Mg	nf

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